FILTERED SAMPLES 5.2

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Filtration is the physical process used to separate the particulate and aqueous fractions of a water sample. Samples are filtered for several purposes; for example, to remove microorganisms in order to help preserve ambient analyte concentrations, to remove suspended materials that interfere with specified analytical procedures, and to determine chemical speciation and fractionation of trace elements for geochemical studies.

Study objectives and the analytes targeted for study dictate the filtration method and equipment to be used. Ambient concentrations of filtered analytes typically can be near the limit of detection; therefore, field personnel must pay strict attention to possible sources of contamination from sampling and processing equipment, construction material of the chamber frame and of the filtration equipment, and the way the equipment is handled. (Equipment and supplies used to filter water samples are described in detail in NFM 2.)

- Check the composition and pore size of the filter medium and the effective filtration area of the filter; these can affect the quality and accuracy of the data and can compromise data-quality requirements.
- To minimize airborne contamination,
  - Filter samples within a processing chamber.
  - Add chemical treatments to samples within a separate preservation chamber.

Filter samples during or immediately after sample collection.

5.2.1 INORGANIC CONSTITUENTS

Most filtration systems currently used by the USGS are appropriate for filtering wholewater samples, if the limitations of each system are taken into account. **Standard USGS procedure is to filter inorganic-constituent wholewater samples through a 0.45-micrometer (µm) pore-size disposable capsule filter.** Filtration through media with pore sizes other than 0.45 µm or with other equipment (such as tangential-flow devices) depends on the use and interpretation of the data and can yield substantially different results for trace-element concentrations.

Data-quality requirements for interpretive studies of ground-water and surface-water chemistry can dictate filtering the sample through a nominal pore size of ≤ 0.2 µm. The quality-assurance procedures used for samples filtered through the 0.45-µm nominal-pore-size capsule, plate, or other filtration equipment also are required for the ≤ 0.2-µm filters. If concentrations of target analytes are analyzed at sub-parts-per-billion levels, more stringent QA/QC measures are needed. Such samples can be filtered through a plate filter or other filtration equipment (for example, a 47-mm-diameter vacuum-filter unit) as long as the equipment used is approved by the study or program, data-quality requirements are met, and additional quality-control samples are collected. For additional information on filtration artifacts, procedures, and equipment, see Kennedy and others (1976), Salonen (1979), McCarthy (1988), McCarthy and Zachara (1989), Puls and Barcelona (1989), Ward and Harr (1990), Horowitz and others (1992, 1994), Williams and others (1993), Robards and others (1994), and Koterba and others (1995).

Cleaning and conditioning of various filter media used for inorganic constituents are summarized in table 5-3. Contamination during sample filtration can be reduced by following the instructions given for cleaning, conditioning, and handling of the filter media.

Table 5-3. Field cleaning and conditioning procedures for media used to filter samples for inorganic-constituent analysis

[µm, micrometer; mL, milliliter; sample, the water to be sampled; µg/L, microgram per liter; mm, millimeter; HNO_3, 1 molar solution of ultrapure-grade nitric acid; HCl, 1 molar solution of ultrapure-grade hydrochloric acid; nutrients, nitrogen and phosphorus species; DIW, District- or laboratory-produced deionized water of known quality, ASTM Type-1 grade or better; IBW, laboratory-produced inorganic-grade blank water; *N*, normal; >, greater than]

Description	Filter media	Field cleaning/ conditioning	Application
Disposable capsule filter[1] (Polypropylene)	Polysulfone, pleated membrane, 0.45-µm or 0.2-µm pore size	Clean with 1,000 mL DIW and remove residual DIW[2] Condition with 25 mL sample	Major ions and nutrients; trace elements with concentrations > 1 µg/L; radio-chemicals and isotopes
Plate filter — 142 mm (Polycarbonate or acrylic)	Cellulose nitrate, tortuous path (0.45 and 0.1 µm are most commonly used pore sizes)	Clean with 500 mL DIW and extract residual DIW Condition with 100 mL sample	Major ions and nutrients; trace elements if concentrations > about 100 µg/L
Cartridge or hand-pressure filter assembly—47 mm (Polypropylene or fluorocarbon polymer)	Cellulose nitrate, tortuous path (0.45, 0.2, and 0.1 µm are most commonly used pore sizes)	Clean with 100 mL DIW and remove residual DIW Condition with 20 mL IBW or 10 mL sample	Major ions and nutrients; trace elements with concentrations at about 1 µg/L or greater
Cartridge or hand-pressure filter assembly—47 mm (Fluorocarbon polymer)	Polycarbonate (such as Nuclepore), direct path (0.40 and 0.1 µm are most commonly used pore sizes)	Soak in HNO_3 rinse with IBW.[3] Remove residual IBW Condition with 20 mL IBW or 10 mL sample	Major ions and nutrients; trace elements with concentrations at about 1 µg/L or greater

[1]Example: Gelman Sciences 12175 (0.45 µm); 600 square-centimeter filtration area. Other disposable capsule filters are available that have different effective filtration area, media type, and media pore size.

[2]For trace-metal analyses at nanogram-per-liter concentration levels, first acid rinse with 500 mL of 1-*N* HCl (polysulfone membranes cannot withstand HNO_3).

[3]Substitute HCI for HNO_3 if sampling includes nutrients.

- Before filtering, designate one member of the processing team as Clean Hands (CH) and another member as Dirty Hands (DH) if using the CH/DH method (NFM 4).
- Wear appropriate, disposable, powderless gloves throughout the process. Vinyl gloves are adequate for inorganic-constituent sampling.
- Filter the samples within a processing chamber to minimize the possibility of contamination.

5.2.1.A Capsule-Filter Procedure

The capsule filter is a disposable, self-contained unit composed of a pleated filter medium encased in a plastic housing that can be connected in-line to a sample-delivery system (such as a submersible or peristaltic pump) that generates sufficient pressure (positive or negative) to force water through the filter. Filter media are available in several other pore sizes, but 0.45 µm is the pore size used routinely for most studies at this time. The capsule filter is required for most studies when filtering samples for trace-element analysis and is recommended when filtering samples for major-ion or other inorganic-constituent analyses.

The following instructions implement Clean Hands/Dirty Hands (CH/DH) techniques and the other QA procedures that are required for trace-element samples with analyte concentrations at the parts-per-billion (ppb) level and that are recommended as good field practice for all samples.

- The DH team member performs operations that are outside of the processing chamber and the CH team member performs operations inside the chamber. DH and CH must wear appropriate disposable, powderless gloves (gloves).
- Preclean capsule filters (step 5 below) before leaving for the field to save field time.

Fill bottles for filtered samples in this sequence: FA (trace elements) ➜ FAM (mercury) ➜ FA and FU (major ions) ➜ FCC or FCA (nutrients) ➜ FAR and all other samples.

To prepare the work space, sample bottles, and capsule filter:

1. CH/DH: Put on one or several layers of gloves.
2. *CH*: Assemble processing chamber, attach chamber cover, and change gloves. Place capsule filter and sample bottles into chamber, and run discharge end of peristaltic pump tubing into the chamber. Open DIW[7] container and cover it with a plastic bag to prevent contamination from airborne particulates.
3. *CH/DH*: (*CH*) Insert intake end of peristaltic pump tubing through the plastic covering and into a l-L container of DIW.
   a. (*DH*): Attach tubing to peristaltic pump head and pump DIW to fill tubing.
   b. Discharge waste rinse water through a sink funnel or a toss (waste) bottle.
4. Discard DIW stored in DIW-prerinsed sample bottles. If sample bottles were not DIW-prerinsed by field personnel:
   a. Wearing gloves, rinse off exterior of each bottle.
   b. Pour DIW into bottle until about one-tenth full.
   c. Cap bottle and shake vigorously about five times.
   d. Uncap and empty bottle.
   e. Repeat b–d of step 4 twice (for a total of three times).
   f. Recap bottles until ready to field rinse.

[7]Office of Water Quality Technical Memorandum 92.01 describes the quality required of the deionized water.

5. **Clean the capsule filter.** If the capsule filter was precleaned, go to the sections that follow on "To filter a composite sample" or "To filter a pumped sample," as appropriate. The steps below comprise sufficient precleaning of the filter for inorganic analytes at the parts-per-billion (ppb) concentration level. More rigorous precleaning procedures that include rinsing with trace-metal-grade hydrochloric acid are required for samples containing ppb concentrations of target analytes (table 5-3). **Only CH touches those portions of tubing that will be in direct contact with the DIW or capsule filter.**

   a. *CH*: In the processing chamber, remove capsule filter from protective bags.

      - Attach pump tubing to inlet connector of capsule filter, keeping tubing as short as possible. **Make sure direction of flow through capsule filter matches the direction-of-flow arrow on the side of the capsule.**
      - To help minimize aeration of the sample (usually for ground-water samples), secure a short length of clean fluorocarbon polymer tubing onto capsule filter outlet to extend into the sample bottle so the bottle can be filled from the bottom up.

   b. *CH/DH*: Pump 1 L of DIW through capsule filter; discharge waste rinse water through a sink funnel or to a toss bottle.

      - *DH* operates the pump at a low speed.
      - *CH* inverts the capsule filter so the arrow on the housing is pointing up. (This expels trapped air from the capsule during initial filling; do not allow water to spray onto the chamber walls.)

   c. *DH*: Remove tubing from DIW reservoir and continue to operate pump in forward mid-range speed position to drain as much of the DIW that remains in the capsule filter as possible. While pump is operating, shake capsule filter to help remove any entrained DIW.

   d. *CH*: Detach capsule filter from peristaltic pump tubing, put it into a clean, sealable plastic bag, and place in a corner of the processing chamber until ready for use.

Filtration procedures differ somewhat, depending on how the sample is collected. If the sample is collected using discrete collection equipment, such as the surface-water bag or bottle sampler or ground-water bailer, use the procedures described below in "To filter a composite sample." If the sample is collected by pumping it directly from the source, use the procedures described below in "To filter a pumped sample." Ground-water samples usually are not collected as a composite. If samples are to be withdrawn from

a well using a bailer, consider using a bailer to which the capsule filter or other filtration device can be connected inline to the bailer bottom-emptying device. Pouring a sample from the top of the bailer into another receptacle aerates the sample and therefore is not a generally recommended procedure for processing ground-water samples.

To filter a composite sample (generally for surface water):

1. Field rinse peristaltic pump tubing with the water to be sampled.
   a. *CH:* Rinse the outside of each end of the peristaltic pump tubing.
   b. *CH*: Transfer intake end of peristaltic pump tubing into composite sample. If a churn splitter is used, transfer intake end of peristaltic pump tubing through churn funnel and reseal plastic bag around the tubing.
   c. *DH*: Start peristaltic pump to slowly pump sufficient sample to completely fill pump tubing.
   d. *CH*: Discard rinse water through the sink funnel or into a toss bottle or other receptacle and dispose of appropriately. Prevent water from ponding in the processing chamber.
   e. *DH*: Stop peristaltic pump after tubing is field rinsed.
2. Field rinse capsule filter:
   a. *CH*: Remove cleaned capsule filter from plastic bag and attach discharge end of the peristaltic pump tubing to the inlet connector on the capsule filter.
      - A clean, small plastic hose clamp may be used to secure the discharge end of the tubing to the capsule filter inlet connector.
      - Check that the direction of sample flow through the capsule filter matches the direction of the arrow on the capsule.
   b. *DH:* Operating the pump at low speed, pump sample through the tubing to the capsule filter.
   c. *CH:* Turn capsule filter so that the outlet is pointing up (arrow on capsule housing is pointing up) and flow of the sample forces trapped air out of the capsule filter while it is filling. **Do not let sample spray onto chamber cover.**
      - The chamber cover must be changed if sample has sprayed onto it.
      - If some water that sprayed onto the chamber cover has dripped into the sample bottle, discard the bottle, change the cover, and collect a new sample.

d. *DH:* Stop the peristaltic pump as soon as the capsule filter is full of sample and all air in the capsule filter has been expelled.

> TECHNICAL NOTE: The goal is to minimize clogging the filter medium with suspended materials by minimizing the volume of sample that will be used to field-rinse the filter.

1. Collect sample filtrate.
   a. *CH*: Check that there is a tight connection between the pump tubing and the capsule filter.

      DH: Check that the intake tube is properly inserted in the sample and start the pump.

      CH: Collect a maximum of 25 mL of the water to be sampled as it discharges through the filter. **Do not exceed 25 mL**.

      CH: Field rinse a precleaned 250-mL FA bottle for trace-element sample only with sample filtrate.

      DH: Stop the pump in time to prevent losing filtrate to waste.

      CH: Cap bottle, shake vigorously, and then discard rinse water into appropriate receptacle.

   b. *DH:* Start pump and resume flow from pump to the filter.

      CH: **Filter only the next 200 mL of the sample** into the trace-element FA bottle (fill to top of upper lip of standard 250-mL polyethylene bottle). Cap bottle securely and set aside for chemical treatment.

   c. *DH:* Stop the pump after the trace-element FA bottle is filled.

   d. If a filtered mercury sample is required, restart pump and repeat steps 3a–c, substituting a FAM bottle for the FA bottle.

   e. *CH:* Field rinse any remaining sample bottles for inorganic analyses. **Use no more than a total of 100 mL of filtrate per capsule filter to field rinse any remaining bottles for filtered sample.**

   f. Fill remaining bottles in the following order: (1) major cations, (2) nutrients and major anions (including alkalinity), (3) radiochemicals (Appendix A5-A), and (4) stable isotopes. Cap each bottle immediately after filling.

To filter a pumped sample (usually ground water):

Ground-water samples usually are withdrawn from a well by means of a submersible pump. Note that this method might be appropriate for some surface-water samples. The capsule filter or other filter assembly is connected inline with the sample tubing in order to collect samples directly from the well.

- When sampling ground water, DH should check that the turbidity values recorded at the end of purging have remained stable. Equipment changes or adjustments that disrupt sample flow can affect sample turbidity and should be avoided. If sample flow is disrupted, pump for several minutes until ambient turbidity values are reestablished.
- **Maintain a smooth, uniform flow.** Do not stop pump or divert flow from capsule filter or other filter assembly during bottle field rinse or filtration, if possible.

  TECHNICAL NOTE: If using a three-way valve, changing the setting to divert the flow of sample being pumped to the filter with a submersible pump can cause air bubbles to form, can air-block the filtration equipment, and can cause changes in pumping rate that could result in increased turbidity. These effects should be avoided to preserve sample integrity; therefore, flow to the filter should not be stopped until all filtration is complete.

1. Field rinse the capsule filter with sample water:
   a. *CH:* Ensure that the sample line is full of sample and free of bubbles; then attach the discharge end of the sample line to the inlet connector on the capsule filter.
      - Practice your technique for attaching the capsule filter to the tubing carrying flowing water so that water does not spray onto chamber walls.
      - Check that the direction of flow matches the direction of the arrow on the capsule.

   b. *DH:* Adjust the sample flow through the sample line to the capsule filter, keeping a slow rate of flow.

   c. *CH*: Turn the capsule filter so the outlet is pointing up (arrow on capsule housing is pointing up) and the flow of sample forces trapped air out while the capsule filter is filling.
      - Do not allow water to spray onto chamber walls.
      - The capsule filter should be full of sample. No air should be left in the capsule filter.

d. Field rinse bottles for inorganic-constituent filtered samples with sample filtrate (section 5.0.3). Use bottles that were already rinsed three times with DIW. Determine whether the potential clogging of pores in the filter medium is of concern for your samples (see TECHNICAL NOTE below).

   CH: Fill a 250-mL FA bottle for trace elements with 25 mL of sample filtrate; cap, shake vigorously, and discard rinse water into appropriate receptacle.

   CH: Fill a FA bottle for trace elements with about 200 mL of sample filtrate (to top of upper lip of 250 mL bottle). Cap bottle and set aside for chemical treatment.

   CH: If a mercury sample is required, field rinse and fill a FAM bottle using the same procedure as for the 250-mL FA bottle.

   CH: Field rinse remaining bottles, trying to use no more than an additional 100 mL of sample filtrate.

   TECHNICAL NOTE: Depending on sample turbidity and composition, the nominal pore size of filter media tends to decrease as the volume of sample passed through the filter increases because pores are clogged by sediment loading or mineral precipitation on the filter (Horowitz and others, 1994). Ground water with turbidity ≤5 NTU should not affect filter pore size appreciably. To minimize the chance of filter clogging, limit the volume of sample passed through the filter by eliminating the field rinse—be sure that you use clean bottles and fill them one after the other. For ground-water sampling, do not stop the pump during the field-rinse and sampling process.

e. *CH:* Collect sample filtrate immediately into any remaining bottles in the following sequence (flow rate should be slow enough to avoid splashing sample out of the bottle): (1) major cations, (2) major anions and nutrients (including alkalinity sample for field titration), (3) radiochemicals (check Appendix A5-A for bottle-rinse and filtration requirements), (4) stable isotopes.

f. *CH*: Cap each bottle immediately.

Rinse FA, FU, FAM, FCA, and FCC bottles with filtered sample—not with raw sample.

After collecting filtered samples:

1. *CH*: If samples require chemical treatment, place FA bottles in the preservation chamber and go to section 5.4.
2. For filtered samples that do not require chemical treatment:
   a. *CH*: Set samples outside processing chamber.
   b. *DH*: Check that information on the bottle label is correct and complete.
   c. *DH:* Pack samples that require chilling in ice or refrigerate immediately.
   d. *DH:* Pack remaining samples for shipping (section 5.5).
3. Rinse all reusable equipment with DIW immediately—before equipment dries.
   - *CH*: If equipment will be reused at another site before returning to the office, rinse immediately with DIW and field clean tubing and other sample-wetted parts of the equipment using the prescribed cleaning procedures (NFM 3).
   - *CH*: If equipment or tubing will not be reused before returning to the office, rinse immediately with DIW and store rinsed tubing and equipment in plastic bags for office or laboratory cleaning.
4. **Discard the capsule filter after filtering each sample—do not reuse.**
5. Document the filtration procedures used on field forms and in field notes.

Use of the 0.45-µm disposable capsule filter for trace-element samples is required for many USGS programs.

5.2.1.B Plate-Filter Procedure

The filtering procedure using a 142-mm-diameter plastic plate-filter assembly is described below. The procedure remains basically the same for plate-filter assemblies of different diameters.

Prepare and precondition plate-filter assembly:

The following instructions pertain to either a 142-mm-diameter or a 47-mm-diameter plastic plate-filter assembly and require that the assembly components have been rigorously cleaned (NFM 3). To avoid recleaning in the field, prepare a set of filtration equipment for each well or surface-water sampling station. (Ignore Step 3 below if plate-filter assembly has been rinsed in the office.)

1. *CH*: Put on gloves. In a processing chamber, open a clean plate-filter assembly and load with the filter.
   a. Using nonmetallic forceps, place the bottom retaining screen on the base of the filter assembly. **Do not interchange bottom and top retaining screens.**
   b. Place the filter on top of bottom retaining screen using clean, blunt plastic or ceramic forceps. Do not touch the filter with hands (gloved or ungloved).
      - Be sure that only one filter is transferred from its original container directly to the plate of the filter assembly. Take care not to transfer the paper liner that separates each filter.
      - The filter should never be removed from the original container until each is transferred to the plate-filter assembly for use. (Exception: polycarbonate (Nuclepore) filter medium is precleaned with acid solution. If transferring one of these, hold the filter with forceps and rinse off acid with inorganic blank water (IBW) dispensed from wash bottle.)
   c. Using forceps, place the top retaining screen on top of the filter.

      TECHNICAL NOTE: If filtering sediment-laden water, a prefilter can be placed between the filter and the top retaining screen.

   d. Dispense 10 to 20 mL of DIW from a wash bottle onto the filter.

e. Close the plate-filter assembly by aligning the top and bottom plates and lightly tightening the plastic bolts, followed by finger tightening opposite pairs of bolts. **Overtightening can cause the plate-filter assembly to warp and leak.** Check that O-rings are in place before closing the assembly. Change gloves.

2. *DH/CH*: Pass the discharge end of the pump tubing through the hole in the side or top of the processing chamber. **Only the CH team member touches sections of tubing that will be in direct contact with the plate-filter assembly.**
   - Keep tubing as short as practical.
   - Attach a short piece of clean tubing to outlet connector of plate-filter assembly.
3. *DH/CH*: Rinse the plate-filter assembly with DIW, using a peristaltic pump, as follows **(rinsing must be repeated each time a clogged filter is replaced with a new filter)**:
   a. *CH*/*DH*: Place intake end of peristaltic pump tubing into a 500-mL container of DIW. Turn pump on low speed.
   b. *CH*: Open the air-vent valve on top of the plate-filter assembly. Tilt the filter assembly slightly to the side and squeeze the outlet tube closed to force trapped air out through the vent. Release the outlet tube. (Venting trapped air is necessary because air bubbles will reduce the effective filtering area by preventing sample from passing through the filter.)
   c. *CH*: Close valve when top is filled with sample.
   d. *CH*: Pump sample through the plate-filter assembly and discard this field-rinse water through the sink funnel or into the toss bottle to prevent the water from ponding in the bottom of the processing chamber.
   e. *CH*/*DH*: Remove intake end of the pump tubing from the DIW container and continue to pump, draining as much of the remaining DIW from the plate-filter assembly as possible.
4. If using a peristaltic pump to transfer the sample to the processing chamber (go to step 5 if sample delivery is with a submersible ground-water pump):
   a. *CH*: Rinse intake end of the peristaltic pump tubing with the water to be sampled.
   b. *CH*: Transfer intake end of the peristaltic pump tubing into the container of sample. If a churn splitter is used, transfer the intake end through the churn funnel and reseal the plastic bag around the tubing.

c. *CH*: Remove peristaltic pump tubing from the inlet connector of the plate-filter assembly and hold the end of the tubing over the sink funnel or toss bottle.

d. *DH/CH*: Start the peristaltic pump in the forward position at slow speed and pump sufficient sample to fill and rinse all pump tubing. Stop the pump after the tubing is rinsed.

5. *CH*: Attach the discharge end of the peristaltic-pump or submersible pump tubing to the inlet connector of the plate-filter assembly.
   - Keep tubing as short as practical.
   - A clean, small, plastic hose clamp can be used to secure the discharge tubing to the inlet connector.
6. *DH*: Start sample flow to the plate-filter assembly.
7. *CH*: Vent trapped air and rinse plate-filter assembly as instructed in steps 3 b–d above.
   - If using a peristaltic pump, turn pump on low speed.
   - If using a submersible pump, maintain a slow and steady flow rate.
8. *CH*: Rinse appropriate sample bottles once with filtrate. Filter no more than 100 mL of sample for the final rinse of all sample bottles that require rinsing.
9. Filter samples, filling bottles in the following order, as applicable to study objectives and sample designation:
   a. Trace elements

      TECHNICAL NOTE: Study objectives and data-quality requirements govern procedures to be used if the filtered trace-element sample is to reflect concentrations of analytes in true solution (the dissolved fraction). Such interpretive studies of ground-water or surface-water chemistry commonly use ≤0.1-µm filter media and plate-filter assembly or a tangential flow method of phase separation. Note that any deviation from the standard procedure for collecting filtered trace-element samples through the 0.45-µm capsule filter must be documented and reported with the analytical results.

   b. Major cations
   c. Nutrients, major anions, and alkalinity sample
   d. Radiochemicals
   e. Isotopes

10. *CH*: If the filter medium clogs before the needed volume of water is filtered, carefully remove the filter and replace with a new filter. Repeat steps 1 through 7. Cap each bottle immediately after filling.
11. **If samples require chemical treatment ⇨Go to section 5.4.**
12. *DH*: After filtration,
    a. Check that information on the bottle label is complete and set the samples aside for shipping (section 5.5). Samples that must be chilled need to be refrigerated or packed in ice as quickly as possible and maintained at 4°C without freezing.
    b. Disconnect and disassemble the plate-filter assembly. **Discard the used filter.**
    c. Rinse all equipment with DIW immediately after use and before it dries. Equipment that has dried after sampling without being rinsed or cleaned needs to be cleaned vigorously with a detergent and rinsed with DIW before the next use. Nonmetallic equipment must also be acid rinsed.
    d. Put rinsed tubing in a plastic bag for cleaning at the office laboratory.
    e. If equipment is to be used at the next site, field clean all the equipment using the procedures described in NFM 3. Field cleaning between sampling sites is carried out while still at the sampling site.
13. Document on field forms and in field notes any modifications to the filtration procedures used.

ORGANIC COMPOUNDS 5.2.2

Standard procedure for phase separation of general trace-organic compounds involves the use of a stainless steel or aluminum 142- (or 293-) mm-diameter plate-filter assembly with glass-fiber filter media and a valveless piston or fluorocarbon polymer diaphragm-head metering pump (section 5.2.2.A). Equipment and procedures differ when filtering samples for dissolved and suspended organic carbon (section 5.2.2.C) and optionally for organonitrogen herbicide analyses (section 5.2.2.B). Required conditioning for filter media is discussed below and summarized in table 5-4.

Table 5-4. Field conditioning requirements for media used to filter samples for organic-compound analysis

[mm, millimeter; mL, milliliter; PBW, pesticide-grade blank water; sample, the water to be sampled; methanol, pesticide-grade methanol; DIW, deionized water]

Filtration equipment *Application*	**Construction materials**	**Filter media**	**Filter cleaning and conditioning[1]**
Plate-filter assemblies: 142 or 293 mm *General trace organic compounds*	Stainless steel or aluminum	Glass-fiber filter[2]	Wet with PBW: 10-20 mL (142 mm) or 50-75 mL (293 mm) Condition with 100-125 mL sample
Disposable capsule filter: 25 mm *Organonitrogen herbicides*	Polypropylene	Nylon	Rinse with 10 mL of methanol No conditioning
Pressure filter apparatus: 47 mm *Dissolved and suspended organic carbon*	Stainless steel or fluorocarbon polymer	Silver metal	Rinse with 100 mL PBW or District-prepared organic-grade DIW Condition with 10-15 mL sample

[1]Do not reuse filters.

[2]Use only glass-fiber filters that have been adequately baked.

The procedures for filtering samples for analysis of trace-organic compounds, including volatile organic compounds, pesticides, and base-neutral compounds, are summarized from Sandstrom (1995). CH/DH techniques and associated QA procedures for inorganic analytes with parts-per-billion concentrations are not required for organic analytes but are recommended as good field practices to maintain the integrity of sample chemistry. Field personnel must wear disposable, powderless gloves (gloves). These gloves must be able to withstand any solvents or other chemicals that will be used during sample processing and equipment cleaning. Equipment and supplies used to filter different types of organic compounds are described in NFM 2. Additional information about organic-compound filtration can be found in Ward and Harr (1990), Manning and others (1994), Shelton (1994), and Koterba and others (1995).

Plate-Filter Procedure 5.2.2.A

Read through the procedures described in Sandstrom (1995) and presented in tables 5-4 and 5-5 and in figure 5-1. Obtain the equipment needed (table 5-5), test equipment operation, and collect an equipment blank if needed. Filtering samples for organic-compound analysis inside a processing chamber and using Clean Hands (CH)/Dirty Hands (DH) techniques is not mandatory but is recommended.

Table 5-5. Equipment for filtration of water-sediment samples for determination of organic compounds

[Modified from Sandstrom (1995); FEP, fluorinated ethylene-propylene; mm, millimeter; mL/min, milliliter per minute; L, liter; µm, micrometer; °C, degree Celsius]

Item	Description of equipment
	Container for unfiltered sample. Clean, laboratory-grade glass bottles with fluorocarbon polymer-FEP-lined lids.
	Fluorocarbon polymer-FEP tubing, 6.35-mm outside diameter.
	Union, 6.35-mm tube (Swagelok Company, Solon, Ohio, No. SS-400-6 or equivalent).
	Fluorocarbon polymer-FEP convoluted tubing, 6.35-mm outside diameter (Cole-Parmer Instrument Company, Chicago, Ill., No. L-06486-02 or equivalent).
	Tube fitting, 6.35-mm diameter tube to 6.35-mm diameter pipe thread (Swagelok Company, Solon, Ohio, No. SS-400-1-4 or equivalent).
	Pump, ceramic-piston, valveless, with 12-volt direct current motor, capable of pumping from 0 to 500 mL/min (Fluid Metering, Inc., Oyster Bay, N.Y., Model QB-1 CSC or equivalent).
	Battery, 12-volt direct current.
	Tube fitting, 6.35-mm diameter tube to 9.53-mm diameter pipe thread (Swagelok Company, Solon, Ohio, No. SS-400-1-6 or equivalent).
	In-line plate-filter assembly, aluminum (or stainless steel), 142-mm diameter (Geotech Environmental Equipment Inc., Denver, Colo., No. 0860 or equivalent).
	Glass-microfiber filter media, binder-free, 142-mm diameter, 0.7-µm nominal pore size (Whatman Inc., Clifton, N.J., GF/F grade, No. 1825C142 or equivalent). Note: The filters must be baked at 400°C for at least 2 hours and kept wrapped in aluminum foil before use.
	Bottle for filtered samples, amber borosilicate glass, 1 L with fluorocarbon polymer-FEP-lined cap.
	Fluorocarbon polymer-FEP squeeze (wash) bottle for organic-grade blank water.
	Stainless-steel forceps for handling the filters.

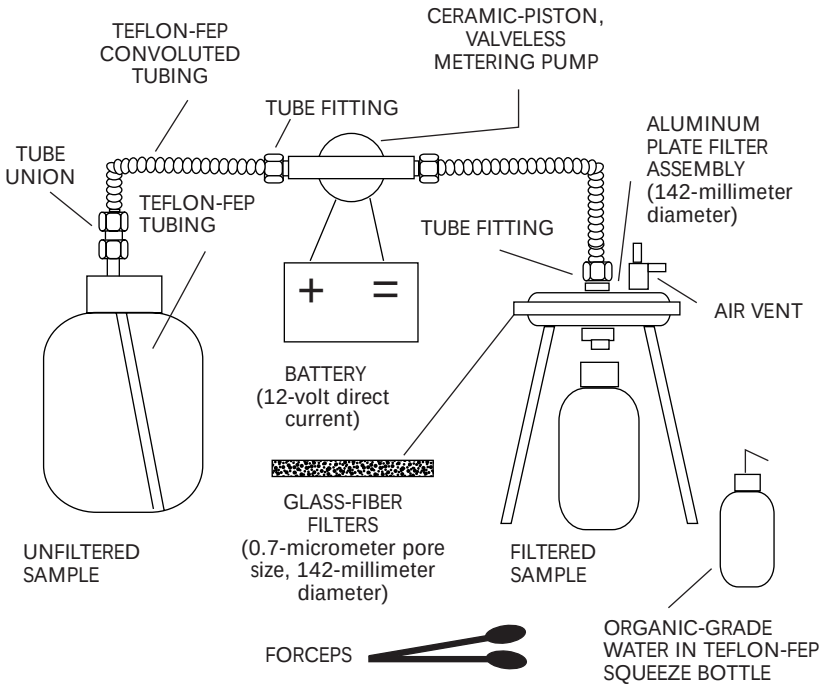

Figure 5–1. An equipment system suitable for filtering samples for analysis of organic compounds (from Sandstrom, 1995).

To filter sample for analysis of general trace-organic compounds in solution:

1. *CH/DH*: Wear appropriate (latex or nitrile) gloves throughout sample processing. Change gloves after setting up equipment. (Wearing several layers of gloves can save time.)
2. *CH*: Load the filter onto the plate-filter assembly within the processing chamber.
   a. Open precleaned plate-filter assembly.
   b. Place one stainless steel support screen on the base of the plate-filter assembly—Use stainless steel forceps.
   c. Place one clean 0.7-µm pore-size glass microfiber filter on top of the screen. **Do not touch the filter with fingers; use stainless steel forceps.**
   d. Wet the filter with a few drops of pesticide-grade blank water (PBW) from a fluorocarbon polymer wash bottle to help keep the filter in place as the unit is assembled.

e. Close plate-filter assembly—Align top and bottom plates. **Lightly tighten** the locking bolts or locking ring. Attach a short length of fluorocarbon polymer tubing to the outlet of the plate-filter assembly to channel filtrate to a toss bottle, sink funnel, or drain.

f. Add 10 to 20 mL of PBW rinse water through the inlet in the upper plate to wet the filter completely before tightening the clamps. (This rinse also helps prevent damage to the filter: a dry filter might rupture when the plate-filter assembly is tightened.)

g. Tighten the locking bolts or ring by hand. **Overtightening can cause the plate-filter assembly to warp and leak and the filter to rupture.**

3. *CH/DH*: Rinse the pump tubing (from a metering pump) or the sample tubing (from a submersible ground-water pump) with the water to be sampled. Discard rinse water into a sink funnel or toss bottle.

4. Set up the pump for filtration.

- *CH*: If using a metering pump, place intake end of tubing into the container holding the sample. Attach discharge end of pump tubing to the inlet connector of the plate-filter assembly. Use a stainless steel compression fitting of the appropriate size to secure the discharge hose to the inlet connector.
- *CH*: If using a submersible pump, attach discharge end of the sample tubing from the pump to the plate-filter assembly, keeping tubing as short as practical. Use a stainless steel compression fitting of the appropriate size to secure the discharge hose to the inlet connector.

5. *CH*: Rinse and condition the filter. The total volume of sample passed through the filter, including rinse water, needs to be accurately determined to ±1 mL and recorded in the field notes.

a. Turn on the metering pump at low speed or open the sample tubing from the submersible pump and operate at a low flow rate.

b. Open the air-vent valve located on top of the plate-filter assembly. Tilt the assembly slightly to the side to allow all trapped air to escape (vent).

c. Close the air-vent valve when water discharges through the valve.

d. Pass 100 mL of sample through the filter to remove any residual liquids from the cleaning or prewetting procedures. If concentration of organic compounds in suspended-material phase is to be determined:

   i. Capture the rinse water in a dry, clean, graduated cylinder.

   ii. Measure and record the actual volume of sample passed through the filter.

e. Discard rinse water to a sink funnel or toss bottle.

6. *DH*: Tare the weight of a clean, baked, glass sample bottle. (First check to see if this is required for the analytical procedures to be used.)
   a. Set up, level, zero, and check the accuracy of the balance with a reference weight. Record accuracy in field notes.
   b. Tare the weight of a dry, clean, capped 1-L amber bottle, and record the weight. Remove the bottle cap.
7. Filter and weigh each sample. (Do not field rinse baked, glass sample bottles.)
   a. *CH*: Resume the flow of sample through the plate-filter assembly.
   b. *CH*: Place the appropriate sample bottle under the outlet of the plate-filter assembly.
   c. *CH*: Collect approximately 1 L of filtered sample for each analytical schedule, but leave headspace in each bottle. If the filter medium becomes too clogged to proceed, go to step 13 below.
   d. *DH*: Cap the bottle(s) and pass sample(s) out of chamber. Wipe the bottle dry with a lint-free laboratory tissue, such as Kimwipe™, to remove any condensation from the outside of the sample bottle.
   e. *DH*: Weigh and record the amount of sample filtered (total weight minus tare weight of bottle).
   f. Chill samples immediately and maintain at or below 4°C without freezing for shipment to the laboratory (section 5.5).

8. *CH*: Remove as much water as possible from the inside of the plate-filter assembly by using the metering pump to pump air through the sample tubing, or by pulling water out through the outlet nozzle with a peristaltic pump, or by using a syringe to apply positive air pressure to the inlet connector. This removes any residual sample and prevents spilling the water-sediment slurry when the plate-filter assembly is disassembled.
9. *CH*: If sediment collected on the filter is to be analyzed for organic compounds:
   a. Carefully disassemble the top of the plate-filter assembly.
   b. Using metal forceps, carefully fold the filter in half and then in half again (quarters).
   c. Transfer the filter to a baked, wide-mouth glass jar with a fluorocarbon-polymer-lined cap.
   d. Record on the jar label and on field forms the total volume of sample that passed through the filter.
   e. Chill and maintain the sediment sample at or below 4°C for shipment to the laboratory (section 5.5)
10. *DH/CH*: If sediment on the filter will not be analyzed, disassemble the top of the plate-filter assembly and remove the filter with forceps. Discard the filter appropriately. Rinse the plate-filter assembly components and tubing immediately after the filter has been removed.
11. *DH/CH*: **If the equipment is to be used at a subsequent site, field clean all equipment while equipment is still wet and before going to the next site.** Clean with detergent solution, rinse with DIW, and final rinse with methanol—do not use methanol on equipment used for TOC, DOC, or SOC samples (NFM 3). If the plate-filter assembly will not be reused before returning to the office, rinse all components with DIW. Put rinsed components and tubing in a resealable bag for cleaning at the office laboratory.
12. Document on field forms and in field notes the filtration procedures used.

13. **If the filter medium becomes clogged before the required volume of sample has been collected,** stop the metering pump or divert the sample flow from the submersible pump (see TECHNICAL NOTE below) and replace the filter with a new filter as indicated in steps a through f below.

> TECHNICAL NOTE: Diverting the flow of sample being pumped with a submersible pump by use of a three-way valve can result in a temporary increase in turbidity (NFM 4). Allow turbidity to clear after reestablishing flow through the sample tubing and to the plate-filter assembly.

a. Remove as much water as possible from inside the plate-filter assembly. The stainless-steel or aluminum plate-filter assembly does not have an upper support screen, so the filter cannot be backflushed. Remove the inlet tubing to the metering pump from the sample and either attach tubing from a peristaltic pump to the outlet and pull residual water out, or use a syringe to apply positive air pressure to the inlet connector.

b. Remove the clogged filter with forceps. **If sediment collected on a filter is to be analyzed for organic compounds, follow directions in step 9**.

c. Load the plate-filter assembly with a new filter and reassemble the unit as described in step 2.

d. Prepare the filter as described in steps 2f and 5a–d, allowing the first 125 mL of sample to remove any sediment particles that may have moved below the filter during the replacement procedure. Use a graduated cylinder to measure volume.

e. Record the volume of sample rinsed through the plate-filter assembly if sediment collected on the filter is to be analyzed for organic compounds. Volume accuracy should be ± 1 mL.

f. Place a tared sample bottle under the plate-filter assembly outlet, resume the flow of sample through the filter, and continue to collect the sample filtrate.

Capsule-Filter Procedure for Processing Samples for Analysis of Organonitrogen Herbicides (Optional) 5.2.2.B

The capsule-filter procedure for filtering samples for organonitrogen-herbicide analysis described below is provided if the option to process these samples onsite is selected. The steps that follow are taken from Sandstrom (1995), which includes more detailed instructions and description of the equipment, including the 25-mm-diameter disposable nylon-media filter capsule (nylon filter):

1. Before leaving for the field site, clean the nylon filter.
   a. Put on appropriate, disposable, powderless gloves (gloves).
   b. Place intake end of the metering pump tubing into the methanol.
   c. Pump about 10 mL through the nylon filter to a used-methanol disposal container.

CAUTION: Do the following if using methanol or other organic solvent:

- **Work under a fume hood or in a well-ventilated area, NOT in the field vehicle.**
- **Wear protection against skin and eye contact and do not inhale fumes.**
- **Collect methanol rinse waste into proper disposal containers and dispose of according to local regulations.**

2. At the field site, cover the field bench or table with a sheet of aluminum foil or Teflon™ to prepare a clean work surface.
3. Place equipment and supplies on the clean work surface. Remove foil or other wrapping from precleaned equipment. Change gloves.
4. Remove the nylon filter from the plastic bag. Rinse the discharge end of the pump tubing with methanol. Discard used methanol to a proper waste container. Attach the metering-pump tubing to the capsule inlet; keep tubing as short as possible.
5. If filtering with a metering pump, transfer the intake end of the pump tubing to the sample. If using a submersible pump to collect the ground-water sample, redirect the sample flow to and from the nylon filter as needed, using a manifold flow-valve system.
6. Purge air from the sample tubing. Before connecting the nylon filter, allow ground-water sample to flow through the tubing at a very low rate. This will require just a few milliliters of sample if a metering pump is used. With sample flowing, connect tubing to the nylon filter. (Use a Luer™ connector of appropriate size to secure the discharge hose to the inlet connector.)
7. Collect at least 100 mL of filtrate in a 125-mL baked amber glass sample bottle. Do not completely fill the bottle. Allow 2–3 cm of headspace. The headspace leaves space for matrix spike standards to be added (if required) and prevents sample loss if the sample freezes.
8. If the nylon filter medium becomes clogged before a sufficient amount of sample has been filtered, replace it with a new nylon filter and repeat steps 6 and 7 until at least 100 mL have been collected.
9. When filtering is complete, cap the bottle firmly. Chill and maintain the sample at or below 4°C without freezing during storage and shipment to the laboratory (section 5.5).
10. Discard the nylon filter. Field clean the pump and tubing as described in NFM 3 before using the equipment at the next site.
11. Document on field forms and in field notes the filtration procedures used.

Procedures for Processing Samples for Carbon Analysis 5.2.2.C

Standard methods are described in this section for processing a sample for analysis of (1) total particulate carbon (TPC), particulate inorganic carbon (PIC), and particulate organic carbon (POC)[1]; and (2) dissolved organic carbon (DOC). The specific method to be used depends on the target analyte and the choice of filter type and filtration equipment, which are to be documented on field forms and in field notes.

- **TPC (Total Particulate Carbon), PIC (Particulate Inorganic Carbon), and POC (Particulate Organic Carbon).** Filtration of the sample requires a 25-mm glass-microfiber filter (see *UPDATE* below). Particulate organic carbon is determined by subtracting the laboratory-analyzed concentrations of particulate inorganic carbon from total particulate carbon; that is, POC = TPC - PIC.
- **DOC (Dissolved Organic Carbon).** Filtration of the sample requires either a disposable capsule unit with a polysulfone pleated filter or a 25-mm glass-microfiber filter (GF/F) (see *UPDATE* below).

> ***UPDATE:*** The NFM-5/99 version of this section (5.2.2.C) entitled "Gas-Pressurized Filter Procedures for Processing Samples for Analysis of Dissolved and Suspended Organic Carbon," which was based on a field method using silver filters, has been moved to Appendix A5-D. That method is no longer used in USGS studies as a standard procedure because of the decreasing availability of the silver filters (Office of Water Quality Technical Memorandum 2000.08).

[1]POC, determined by a calculation, is distinguished from the suspended organic carbon (SOC) analysis, which is determined by direct analysis of organic carbon residue on a silver filter. USEPA method 440.0 is used for laboratory analysis of the TPC and PIC samples and also provides direct determination of total particulate nitrogen (TPN) concentration.

Equipment and equipment-cleaning procedures

The equipment needed to process samples for analysis of TPC and PIC depends on whether the pressure-filtration method (table 5-6a) or the vacuum-filtration method (table 5-6b) will be used. The equipment options for processing samples for analysis of DOC are given in table 5-6c. (Refer to Appendix A5-D if the silver-filter method will be used to process samples for analysis of TOC, SOC (suspended organic carbon), or DOC.)

Equipment should be cleaned while still wet from sampling, preferably before leaving the field site. Immediately after each use, rinse the carbon-processing equipment at least three times with DIW and store it in a plastic bag until sampling is complete and there is time to clean the equipment using USGS standard procedures.

- Clean the carbon-collection and carbon-processing equipment according to the standard procedures described in NFM 3.3.4.C. **Do not use methanol or any other organic solvent to clean this equipment** (see TECHNICAL NOTE).
- If it is necessary to return to the office before cleaning the equipment, be sure to field rinse the equipment onsite immediately after use and then place it in a clean plastic bag for transport.
- After the equipment has been cleaned, double-wrap all apertures and the filter apparatus with aluminum foil and store them inside a sealable plastic bag for transport to the next site or storage in the office.

> TECHNICAL NOTE: Periodically check the NFM Comments and Errata page (http://water.usgs.gov/owq/FieldManual/mastererrata.html)under Chapters A3 and A5 for an update or any changes in equipment-cleaning procedures. If a circumstance arises in which methanol-cleaned equipment must be used to collect samples for DOC analysis, it is necessary to (1) record and report the total volume of water that has passed through the equipment before the DOC sample is collected, (2) collect a field blank sample for laboratory analysis by passing organic-grade blank water through the equipment, (3) collect a source-water blank for laboratory analysis, and (4) compare the laboratory results of carbon concentrations for the environmental and quality-control samples and document the results in field notes and in any report in which the DOC data are presented.

Table 5-6a. Equipment and supplies used to process samples for analysis of total particulate carbon and particulate inorganic carbon using the pressure-filtration method

[FEP, fluorinated ethylene-propylene; DOC, dissolved organic carbon; mm, millimeter; µm, micrometer; °C, degrees Celsius; mL, milliliter; in, inch; oz, ounce; lbs/in^2, pounds per square inch; mg/L, milligrams per liter; VOC, volatile organic compound]

Item	Description/Comments	Supplier or USGS One Stop Shopping Item Number[1]
FEP pressure-filtration apparatus (DOC-25)	Holds a 25-mm filter	Q444FLD
Ring stand and clamp	Holds the DOC-25 filtration unit	Open market
Filter, in-line vent, 50 mm	0.2-µm pore size; pre-filter to remove airborne particulates	Q445FLD
Peristaltic pump	Adjustable flow rate	Open market
C-Flex tubing	For use with pump	Q432FLD or Open market
Precombusted (baked) glass-microfiber filters (GF/F)	25 mm, 0.7-µm pore size, laboratory-baked at 400°C (3 filters are required)	Q441FLD
Metal forceps, two pair	Standard metal forceps for handling filter media	Q347BACT
Glass cylinder	100-mL graduated cylinder, cleaned	Open market
Aluminum foil squares	6 in x 6 in	Q443FLD
Whirl-Pak bags	6 oz	Q22FLD
Whirl-Pak bags	18 oz	Q21FLD
Aluminum foil	Heavy duty	Open market
Cooler and ice	Standard; check with shipper for size and weight restrictions	Open market
Replacement filter-support screen	25 mm, either stainless steel or polysulfone	Pall Gelman Laboratory Part nos. 79791 or 87265
Pressure gage (optional)	Glycerin-filled, 0-30 lb/in^2, to be inverted into side of a plastic tee that is positioned in-line between the peristaltic pump and the DOC-25 filtration unit.	Cole Parmer catalog no. P-07370-70 or equivalent
Organic grade water (if using this method for DOC processing)	Laboratory analysis of the water must certify a concentration of organic carbon that is less than the long-term laboratory reporting limit for DOC (currently <0.16 mg/L). Check the laboratory analysis for the lot number to confirm that it can be used.	N1590 (Pesticide-grade blank water) or N1580 (VOC-grade blank water)

[1]The equipment designated by the letters Q or N preceding a unique number is supplied exclusively for USGS studies through the USGS internal One Stop Shopping. The USGS performs quality-control checks for such equipment. Such equipment can be obtained for non-USGS studies on the open market or other source specified by the user. "Open market" designates equipment to be obtained from a retail or other vendor.

Table 5-6b. Equipment and supplies used to process samples for analysis of total particulate carbon and particulate inorganic carbon using the vacuum-filtration method

[mL, milliliter; mm, millimeter; µm, micrometer; °C, degrees Celsius; in, inch; oz, ounce; FEP, florinated ethylene-propylene]

Item	Description/Comments	Supplier or USGS One Stop Shopping Item Number[1]
Filtration flask	Polypropylene, 500 or 1,000 mL	Open market
Filter funnel	Polysulfone, 25 mm with 200-mL reservoir[2]	Open market
Peristaltic pump or hand pump	Adjustable flow rate	Open market
C-Flex tubing	For use with pump	Q432FLD or Open market
Baked glass-microfiber filters (GF/F)	25 mm, 0.7-µm pore size, laboratory-baked at 400°C (3 filters are required)	Q441FLD
Metal forceps, 2 pair	Standard metal forceps for handling filter media	Q347BACT
Glass cylinder	100-mL graduated cylinder	Open market
Aluminum foil squares	6 in x 6 in	Q433FLD
Whirl-Pak bags	6 oz	Q22FLD
Whirl-Pak bags	18 oz	Q21FLD
Aluminum foil	Heavy duty	Open market
Cooler and ice	Standard: check with shipper for size and weight restrictions	Open market

[1]The equipment designated by the letters Q or N preceding a unique number is supplied exclusively for USGS studies through the USGS internal One Stop Shopping. The USGS performs quality-control checks for such equipment. Such equipment can be obtained for non-USGS studies on the open market or other source specified by the user. "Open market" designates equipment to be obtained from a retail or other vendor.

[2]The filter-support screen can be replaced with a stainless-steel screen like the one used in the FEP pressure-filtration apparatus. Contact Pall Gelman Laboratory, 600 Wagner Road, Ann Arbor, MI, 48103-9019; phone (734) 665-0651.

Do not use methanol or any other solvent to clean TPC or DOC equipment (NFM 3).

Table 5-6c. Equipment and supplies used to process samples for analysis of dissolved organic carbon

[µm, micrometer; GF/F, glass microfiber filter; mm, millimeter; °C, degrees Celsius; FEP, florinated ethylene-propylene; oz, ounce; mL, milliliter; DOC, dissolved organic carbon; <, less than; mg/L, milligrams per liter; *N*, normal; VOC, volatile organic compound]

Item	Description and Comments	Supplier or USGS One Stop Shopping Item Number[1]
Gelman Sciences Supor capsule filter	Pleated polysulfone filter medium, in disposable polypropylene casing, 0.45-µm pore size	Q398FLD
----------or----------	----------or----------	----------or----------
Precombusted (baked) glass microfiber filters (GF/F)	25-mm diameter, 0.7-µm nominal pore size, laboratory baked at 400°C FEP pressure-filtration apparatus or filtration flask with funnel and associated equipment is required, as indicated in table 5-6a and table 5-6b, respectively	Q441FLD
4-oz amber glass bottle, baked	Bottles (125 mL) supplied for DOC samples have been pre-cleaned and baked at 400°C and quality-controlled to meet a detection limit criterion for organic carbon of <0.1 mg/L	Q28FLD
Sulfuric acid (H_2SO_4) preservative	4.5*N*-H_2SO_4, supplied in 1-mL vials	Q438FLD
Organic-grade water	Laboratory analysis of the water must certify a concentration of organic carbon that is less than the long-term laboratory reporting limit for DOC (currently <0.16 mg/L). Check the laboratory analysis for the lot number to confirm that it can be used.	N1590 (Pesticide-grade blank water) or N1580 (VOC-grade blank water)
Aluminum foil	Heavy duty	Open market
Cooler and ice	Standard; check with shipper for size and weight restrictions	Open market
Foam bottle sleeve	Individual bottles are slipped into foam sleeves to protect from breakage.	Q137FLD

[1]The equipment designated by the letters Q or N preceding a unique number is supplied exclusively for USGS studies through the USGS internal One Stop Shopping. The USGS performs quality-control checks for such equipment. Such equipment can be obtained for non-USGS studies on the open market or other source specified by the user. "Open market" designates equipment to be obtained from a retail or other vendor.

TPC, PIC, and POC sample processing

The sample-processing options described below involve use of either the pressure-filtration or vacuum-filtration method. The equipment and supplies needed are listed in tables 5-6a and 5-6b, respectively. Particulate analytes (TPC, PIC, POC, SOC) are reported in units of mass per volume (milligrams per liter), and therefore **the volume of sample passed through each filter must be measured accurately and recorded on the field form and the Analytical Services Request (ASR) form**.

- The amount of water to be filtered to obtain a sufficient quantity of material for the analysis depends on the suspended-sediment concentration and/or on the concentration of humic and other substances (such as organic and inorganic colloids).
- A graph of the historical stream stage plotted against suspended-materials concentration can aid in estimating concentrations of suspended materials. Suspended-material concentrations can be used to help select the volume of sample to be filtered for a POC determination (table 5-6d).
- Record the filtrate volume passed through each filter used for particulate analysis. **This is critical for calculation of POC concentrations.**

Table 5-6d. Guidelines for selecting the volume needed for filtration of samples for analysis of suspended and particulate organic carbon
[Guidelines are based on sand-sized materials; other physical property factors and chemical composition were not taken into account; mg/L, milligrams per liter; mL, milliliters; >, greater than]

Approximate concentration of suspended materials (mg/L)	Volume of sample to be filtered (mL)
1 - 30	250
30 - 300	100
300 - 1,000	30
> 1,000	10

For TPC and PIC samples, be sure to record the total volume of water that passed through each GF/F filter.

To process samples for analysis of TPC and PIC:

1. **Sampling location and collection:** Study objectives and site characteristics determine where the sample will be collected. Follow guidelines for (1) preventing sample contamination as described in NFM 4.0, (2) using the appropriate isokinetic or nonisokinetic method as described in NFM 4.1, and (3) preparing composites and subsamples or discrete samples as described in NFM 5.0 through 5.1.1.[2] Avoid the use of methanol-rinsed equipment.

2. Select one of the following three options. (Note that the actual volume of sample needed is determined by the concentration of particulates for the specific site and not by the bottle volume.)

   - **Collect a discrete sample with a weighted bottle sampler at centroid of flow** (see NFM 4, section 4.1.1.A, VCF method)—Load the sampler with baked 125-mL DOC bottles or a 1-L baked pesticide bottle, depending on the type of sampler being used. Cap all bottles securely after they are filled with sample.

   - **Collect, composite, and process the sample through a cone splitter**—Using procedures described in 5.1.1.B, collect the TPC/PIC subsample from the methanol-free cone splitter into a baked, 1-L pesticide bottle or into three to four baked 125-mL DOC bottles. Cap all bottles securely.

   - **Collect, composite, and process the sample through a churn splitter**—Using procedures described in 5.1.1.A, collect the TPC/PIC subsample from the churn splitter into a baked, 1-L pesticide bottle or into three to four baked 125-mL DOC bottles. Cap all bottles securely.

     TECHNICAL NOTE: An experiment to test the effect of sand in the polyethylene churn splitter on particulate carbon concentrations showed that, under most sampling conditions, the abrasion of material from the churn by sand particles will result in negligible bias in the analytical results. Caution is recommended in situations where very large concentrations of sand particles coincide with carbon concentrations that are close to the analytical minimum reporting limit (MRL).

[2]The guidelines described were designed for stream sampling. These procedures can be adapted for the collection of TPC, PIC, and TPN in ground-water samples, if necessary.

ALERT! Do not field rinse the baked DOC or pesticide bottles.

3. Cover the bench or table with a sheet of aluminum foil to make a clean work surface. Put on disposable, powderless gloves. Assemble necessary equipment and supplies on the clean work surface.

   a. Fold into thirds the aluminum foil square(s) into which the filters will be placed.

   b. To remove airborne particulates, attach an in-line, 0.2-µm pore-size filter to the inlet side of a dry pump hose between the filtration apparatus and the peristaltic or hand pump.

   c. Attach pump tubing to pump.

   d. Remove the aluminum foil wrapping from the equipment.

   e. Change gloves.

4. Prepare the filtration apparatus.

   • **Pressure-filtration method:**

      a. Open the bottom of the DOC-25 filtration unit.

      b. Using metal forceps, place a 25-mm, 0.7-µm pore size, GF/F onto the support screen in the base of the DOC-25 apparatus.

      c. Push the bottom white ring that holds the filter base up against the filter unit and screw it onto the base of the filtration-apparatus barrel by screwing the blue top ring down onto the bottom white ring.

         – Finger-tighten only. Turning the bottom white ring will cause the outer edge of the filter to be cut off, making removal of the filter difficult.

         – Take care not to wrinkle or tear the GF/F.

      d. Place the DOC-25 apparatus into the ring-stand clamp. Place a bottle or beaker under the DOC-25 filtration unit.

      e. Shake the sample vigorously to suspend all particulate matter and immediately pour an aliquot of the sample into the barrel of the DOC-25 apparatus. While pouring, ensure that the particulates remain suspended.

      f. Screw the top part of the DOC-25 apparatus onto the barrel and attach the peristaltic pump tubing.

- **Vacuum-filtration method:**

  a. Place the filter funnel on the filter flask.

  b. Lift the top part of the filter funnel.

  c. Using metal forceps, place the GF/F onto the base of the filter funnel. **Make sure the filter is not wrinkled or torn.**

  d. Place the top part of the filter funnel back on the base.

  e. Shake the sample vigorously to suspend all particulate matter and immediately pour an aliquot of the sample into the filter funnel. While pouring, swirl sample to ensure that the particulates remain suspended (top of filter flask can be covered with aluminum foil while swirling sample).

  f. Attach the pump tubing from the peristaltic pump or hand pump to the vacuum flask.

5. Apply pressure (pressure filtration) or suction (vacuum filtration) to start the flow of sample water through the filtration apparatus.

   - If using a peristaltic pump to pressurize the DOC-25, install a pressure gage in the line.

     a. Do not exceed about 15 lbs. of pressure.

     b. During pumping, a drop in pressure will signal when the last of the sample water has passed through the filter.

6. After an aliquot of sample has been filtered, tap the bottom of the filter apparatus and increase the pressure slightly to dislodge the remaining drops of sample water. When no more filtrate comes out:

   a. Depressurize the filtration apparatus carefully.

      – **Pressure-filtration method**: Remove the tubing to release the pressure and then remove the top of the DOC-25 apparatus. Check that there is no water on the filter and that the filter is covered with particulates. The particulate cake should not be extremely thick.

      – **Vacuum-filtration method**: Remove the foil cover and look into the top of the filter funnel. Check that there is water on the filter and that the filter is covered with particulates.

   b. If the filter is dry but not covered with particulates, add another aliquot of sample by repeating steps 4e-f, 5, and 6a until the filter is loaded to capacity. **It is important that all the water in the barrel be passed through the GF/F, leaving the filter "dry."**

   c. After the filter is dry and covered with particulates, go to step 7.

7. Pour the filtrate into a graduated cylinder and measure and record the volume on the field form and on the "Comments to NWQL" line of the ASR form.

8. Using organic-grade water, rinse any remaining particles from the sides of the DOC-25 barrel or the sides of the filtration funnel. **Do not include the rinse water in the measured volume.**

9. Discard filtrate. **Do not send this filtrate to the laboratory for analysis of DOC.**

10. After all the organic-grade water filtrate has passed through the DOC-25 filtration unit:

    a. Remove the DOC-25 apparatus from the ring stand.

    b. Continue pumping, rotating the DOC-25 apparatus at a slight angle while tapping the side of the filtration unit to evacuate any remaining water droplets that are clinging to the sides of the filtration unit. This procedure moves droplets toward the center of the filter surface for final passage through the filter.

    c. The procedure is complete when filtrate droplets are entirely evacuated and have passed through the filter-holder funnel.

11. After completing the rinse, depressurize the filtration apparatus. Change gloves.

12. Lift the top off the filter funnel to check that the filter is dry before proceeding to carefully remove the bottom of the DOC-25 apparatus.

    a. Open the previously folded aluminum foil square and place it on the clean work surface.

    b. Gently remove the filter from the filter holder with metal forceps. Do not touch the filter with your fingers. Using two metal forceps:

        i. Place the filter so that it is centered on one of the creases in the aluminum foil square; start the fold with the forceps, then press the foil down on top of the filter to complete the fold.

        ii. Fold it in half with suspended material on the inside. Do not lose any suspended material.

Wear safety glasses when pressurizing or depressurizing a filter apparatus.

13. Repeat steps 4-12 two more times until a total of three filters (two for TPC and one for PIC) have been processed.

    - **If the same volume of sample water was filtered through all three filters,** place them all, side by side, into one aluminum-foil envelope.
    - **If different volumes have been filtered,** use either three separate, properly labeled aluminum foil envelopes or use a single packet and write the volume for each filter on the outside of the foil in which each of the filters is located.

14. Close the other flap of the aluminum foil, turning the ends up carefully.

    i. Label the aluminum foil envelope(s) with site identification, date and time, total filtered volume of sample, laboratory sample-designation code, and the laboratory schedule requested.

    ii. Do not use tape and, if a preprinted label is used, do not let it wrap around the ends of the envelope. (The envelope will be opened and used at the laboratory when drying the filters.)

15. Place labeled aluminum foil envelope(s) into small (6 oz) Whirl-Pak bag(s) and seal the bag(s).

16. Place the 6-oz Whirl-Pak bag(s) inside a large (18 oz) Whirl-Pak bag and seal the large bag.

17. Place the 18-oz Whirl-Pak package into an ice-filled cooler and maintain the samples at or below 4°C during storage and shipment to the laboratory.

For TPC and PIC analyses, record the TOTAL volume of sample that passed through each filter.

DOC sample processing

The sample-processing options described below involve filtering the sample either through a GF/F and pressure-filtration apparatus (the pressure-filtration method), or through a capsule filter (the capsule-filter method) (Office of Water Quality Technical Memorandum 2000.08). The pressure-filtration and capsule-filter methods are described below and the equipment needed for each method is listed in tables 5-6a and 5-6c, respectively. The silver-filter method and equipment are described in Appendix A5-D.

- Use organic-grade water when collecting an equipment blank or field blank for quality control. Organic-grade water is deionized water that has been certified by laboratory analysis of the lot to have an organic-carbon concentration that is less than the laboratory reporting limit (currently at 0.16 mg/L for DOC).
- Each equipment or field blank designated for carbon analysis should be accompanied by a source blank collected from the same lot of organic-grade water as is used for the equipment and field blanks.

  TECHNICAL NOTE: A laboratory study to compare the results of the DOC analysis in same-source water that was filtered through 0.45-µm pore-size silver-filter media, the Gelman 0.45-µm Supor™ capsule filter, and 0.7-µm GF/F determined no statistically significant differences in DOC concentrations (Charles Patton and George Aiken, U.S. Geological Survey, written commun., 2001).

- USGS designations and preservation treatment for various filtered samples are listed below. The general order of preservation is to acidify all samples requiring HCl treatment first, followed by those for H_2SO_4 treatment if nutrient samples are to be acidified, and then those for HNO_3 treatment. Wholewater samples are preserved along with their filtered counterparts. The chamber cover is changed with each change in the acid treatment.

  - FAM: filtered, acidified with HCl, for mercury analysis.
  - FCC: filtered and chilled to ≤ 4 °C for nitrogen and phosphorus nutrient analysis.
  - FCA: filtered, acidified with H_2SO_4, and chilled to ≤ 4 °C for nitrogen and phosphorus nutrient analysis.
  - DOC: filtered, acidified with H_2SO_4, for dissolved organic carbon analysis.
  - FA: filtered, acidified with HNO_3, for trace-element and major-cation analysis.
  - FAR: filtered, acidified with HNO_3, for radiochemical analysis.

Capsule-filter method:

1. Collect samples. Set up a clean capsule filter and sample bottle(s) within a protective chamber using the Clean Hands method (NFM 4.0.1).

   a. **Surface Water:** Follow guidelines for (1) preventing sample contamination as described in NFM 4.0, (2) using the appropriate isokinetic or nonisokinetic method as described in NFM 4.1, (3) preparing composites and (or) subsamples or discrete samples as described in NFM 5.0 through 5.1.1, and (4) equipment selection and quality control as described in the TECHNICAL NOTE below.

   b. **Ground Water:** Follow standard guidelines for (1) well purging (NFM 4.2), (2) sampling (NFM 4.0, 4.2, 5.0, and 5.1.2), and (3) equipment selection and quality control as described in the TECHNICAL NOTE below. Use a clean bailer that has not contacted methanol if other sampling equipment has been methanol-rinsed. Sample collection and filtration can be conducted in the same protective chamber.

   TECHNICAL NOTE: Process the DOC sample after other filtered samples have been processed. To prevent methanol contamination of the sample, do not use methanol-rinsed collection and processing equipment, and use a new capsule filter. Collection and analysis of field-blank and source-blank samples is recommended. If methanol-rinsed equipment must be used, collection of these blanks to correlate with each DOC sample is required, regardless of the volume of water passed through the system before DOC sample collection. Taking these quality-control measures does not remove the possibility of methanol contamination of the sample, however.

2. Change gloves. Place a 125-mL baked glass amber bottle under the capsule filter outlet.

   - Do not field rinse the DOC bottle.
   - Do not splash sample water.
   - Pass 1 L of **organic-grade water** (certified for organic-carbon concentration of less than the laboratory reporting limit through the capsule filter). If collecting a quality-control sample, go to step 3.
   - Pass at least 1 L of **sample water** through the capsule filter before collecting the DOC sample.

3. Fill the bottle to its shoulder.

4. Cap the bottle and transfer it to the preservation chamber.

5. Change gloves. Open the DOC bottle in the preservation chamber. Add the contents of a 1-mL H_2SO_4 vial to the DOC sample.
6. Cap the DOC bottle securely. Shake the sample bottle vigorously to mix the sample and H_2SO_4.
7. Remove the DOC bottle from the preservation chamber.
8. Check that the bottle is labeled correctly and completely. Place the bottle in a foam sleeve and then into an ice-filled shipping container.
9. Maintain the sample at or below 4°C without freezing (NFM 5.5).

Pressure-filtration method:

1. Collect sample(s).
   a. **Surface Water:** Follow guidelines for (1) preventing sample contamination as described in NFM 4.0, (2) using the appropriate isokinetic or nonisokinetic method as described in NFM 4.1, (3) preparing composites and (or) subsamples or discrete samples as described in NFM 5.0 through 5.1.1, and (4) equipment selection and quality control as described in the TECHNICAL NOTE below.
   b. **Ground Water:** Follow standard guidelines for (1) well purging (NFM 4.2), (2) sampling (NFM 4.0, 5.0, and 5.1.2), and (3) equipment selection and quality control as described in the TECHNICAL NOTE below.

TECHNICAL NOTE: To prevent methanol contamination of the sample, avoid using methanol-rinsed collection and processing equipment. If methanol residue is a concern, process the DOC sample either before introduction of any methanol-rinsed equipment or well after the work area has been cleared of methanol-rinsed equipment and methanol fumes. Collection and analysis of field-blank and source-blank samples is recommended. If methanol-rinsed equipment must be used, collection of these blanks to correlate with each DOC sample is required, recording the volume of water passed through the system before DOC sample collection. Taking these quality-control measures does not remove the possibility of methanol contamination of the sample, however.

2. Cover the bench or table with a sheet of aluminum foil to make a clean work surface. Assemble the necessary equipment on the clean work surface, wearing disposable powderless gloves.

   a. When using the DOC-25 filtration apparatus, remove airborne particulates as follows: attach an in-line, 0.2-µm pore-size filter (table 5-6a) to the inlet side of a dry pump hose or to the outlet between the peristaltic pump and the DOC-25 unit. If attaching the DOC-25 on the inlet side, be sure to dedicate a piece of tubing for the sole purpose of channeling air flow.

   b. Remove the aluminum foil wrapping from the precleaned equipment.

   c. Change gloves.

3. Prepare the filtration apparatus:

   a. Remove the bottom barrel of the filtration apparatus.

   b. With metal forceps, place a clean GF/F filter onto the support screen in the base of the filtration apparatus. **Make sure that the filter medium is not wrinkled or torn.**

   c. Push the bottom white ring that holds the filter base up against the filter unit and screw it onto the base of the filtration-apparatus barrel by screwing the blue top ring down onto the bottom white ring.

      – Finger-tighten only. Turning the bottom white ring will cause the outer edge of the filter to be cut off, making removal of the filter difficult.
      – Take care not to wrinkle or tear the GF/F.

   d. Open the top of the filtration-apparatus barrel and fill with approximately 100 mL of wholewater sample.

      – **For water with high concentrations of suspended materials** (usually in surface water), collect the sample into a clean baked glass bottle, cap it securely, place it on ice, and allow the suspended materials to settle; then, pour 100 mL of the clear supernatant into the filter barrel.
      – **For surface water,** the 100-mL wholewater sample can be either a subsample collected from the churn or cone splitter or the supernatant from the bottle(s) used in the weighted-bottle sampler.
      – **For ground water,** the 100-mL wholewater sample is collected directly from the pump discharge unless turbidity is high. For turbid samples, follow the procedure described above for water with high concentrations of suspended materials.
      – **For a quality-control sample,** use organic-grade blank water.

e. Screw the top part of the filter apparatus onto the barrel and attach the pump tubing.

f. Apply pressure, regulated to less than 15 lb/in^2, to start the flow of sample water through the filter apparatus.

g. Place a 125-mL clean, baked glass bottle under the discharge tube of the filtration apparatus. **Do not field rinse the DOC bottle.**

h. Fill the DOC bottle to the shoulder with sample filtrate.

- If the filter clogs before 100 mL of sample for DOC analysis can be filtered, depressurize the filtration unit, empty the remaining volume of wholewater sample from the barrel, and remove the clogged GF/F filter.
- Using clean metal forceps, replace the old filter with a new GF/F filter, following the directions from steps 3b-g above for a pressure-filtered DOC sample.

4. After the DOC sample bottle has been filled to the shoulder, cap the bottle and transfer it to the preservation chamber.

5. Depressurize and dismantle the filtration apparatus, discarding the used GF/F filter(s). Clean the apparatus immediately (while still wet), following the USGS procedures prescribed in NFM 3.3.4.C. If the apparatus cannot be field-cleaned immediately, it must be cleaned the same day it is used and before being reused—rinse it copiously with DIW and place it into a plastic bag so that it will not dry before being cleaned.

6. Change gloves before working in the preservation chamber.

7. In the preservation chamber, open the DOC bottle. Add the contents of a 1-mL vial containing 4.5*N* H_2SO_4 preservative.

8. Cap the DOC bottle securely and shake vigorously to mix the sample. Remove the DOC sample bottle from the preservation chamber.

9. Check that the bottle is labeled correctly and completely. Place the bottle into a foam sleeve and into an ice-filled shipping container (see NFM 5.5 for correct shipping procedures).

10. Maintain the sample at or below 4°C without freezing (NFM 5.5).

Wear safety glasses when pressurizing or depressurizing the filter apparatus.

11. To collect a QC sample:

    a. Use the same carbon-processing equipment after it has been cleaned (see step 5, above).

    b. Label bottles.

    c. Change gloves.

    d. Working in a clean processing chamber, process a sample of organic-grade water through the cleaned carbon-sampling and carbon-processing equipment, following the steps prescribed in steps 2-4 above. Cap securely and pass the bottle to the preservation chamber.

    e. Follow steps 6-10 above for sample preservation, handling, and shipping.

    f. Depressurize and dismantle the filtration apparatus, discarding the used filter(s). Allow the apparatus components to air dry in a clean chamber. Cover the apertures of the dry apparatus with aluminum foil and place in a clean, sealable plastic bag for storage.

Never increase the pressure in a filter apparatus to greater than 15 lb/in^2 in order to increase the rate of filtration.